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## PLATINUM IN NORTH CAROLINA.

By PAUL R. HEYL.

(Read February 7, 1913.)

North Carolina, on account of the variety of different minerals it affords, may well be classed with Freiburg, Saxony, and Franklin, N. J. More than one hundred and eighty different minerals occur there, and in some cases minerals otherwise rare occur there in commercial quantities.

The gold mines of North Carolina have been known for a century, and it would be but natural to expect small quantities of platinum to be found in such localities. The first announcement to this effect was published by Shepard in 1847.<sup>1</sup> It appears from his report that a nugget of platinum weighing 2.5 grains was found among the gold washings in a rocker at Mr. Erwin's mine in Rutherford county. The miner who picked it out supposed it to be silver, and other miners in the vicinity claimed to have seen similar lumps occasionally. It would seem, however, that such occurrences must be very exceptional, as Hidden,<sup>2</sup> in 1881, failed to find any platinum in five localities, and Venable in 1892,<sup>3</sup> after a careful examination of gold washings from several places and a failure to find any platinum therein, was inclined to distrust Shepard's report.

In 1894,<sup>4</sup> however, Hidden discovered sperrylite, the native arsenide of platinum, in panning gravel from a creek in Macon county, and traced it to its source in rock on the top of a mountain.

The present communication deals with the occurrence of platinum in a different part of the state, and is believed to be the first published notice of the subject. The interest centers around the little village of Ruffin, in Rockingham county, about fifteen miles south of Danville, Virginia. The country hereabouts is devoted mainly to farming, tobacco being the chief crop. Apart from a

<sup>1</sup> *American Journal of Science*, series 2, Vol. 4, p. 280, 1847.

<sup>2</sup> *Ibid.*, series 3, Vol. 22, 1881, p. 25.

<sup>3</sup> *Ibid.*, series 3, Vol. 43, 1892, p. 540.

<sup>4</sup> Kemp's "Report on Platinum," *Bull. U. S. Geol. Survey*, No. 193.

granite quarry a few miles south of the village no form of mining is practised in the neighborhood, although some fifty miles farther south, near Cedar Falls, gold is extracted from a rock deposit in which it occurs rather irregularly.

The beginning of the platinum story goes back some fifteen or sixteen years. At that time, according to neighborhood tradition, the son of a Mr. Harralson, a landowner in the vicinity, picked up in the creek a stone which attracted him by the yellow crystals scattered through it. Supposing it to be gold, he showed it to his father, who sent some of the rock for examination to a Mr. Wilson, at that time chemist for a phosphate concern in Baltimore. Mr. Wilson saw at a glance that the supposed gold was pyrite, but thought it worth while to see if the pyrite was auriferous, and directed his assistant, a Mr. Walsh, to make some assays. Mr. Walsh found no gold, but encountered something else which puzzled him for a while, until he finally obtained a reaction for platinum.

The quantities of platinum shown by this sample of rock were surprising. Four assays in Mr. Wilson's laboratory gave the following figures in ounces per ton: 4.76, 2.40, 2.85, 3.60. More rock was sent up at Mr. Wilson's request, and four more assays were made, all of which were blank. Greatly incensed at what he supposed a trick, Mr. Wilson visited the ground and nearly came to blows with Mr. Harralson. Finally convinced of the latter's sincerity, Mr. Wilson proceeded to investigate the matter on a large scale. He turned the creek out of its bed and put down a charge of dynamite. Samples of this rock again showing platinum, a car load was sent to the Mecklenburg Iron Works, in the same state, and there ground and washed on a Wilfley table. The following results were obtained:

Concentrates 30 to 1.
Ounces Per Ton in the Concrete.
97.2
18.0
61.2
42.0
10.2
24.6
25.2
42.0

Mr. Wilson also put down a hole in the ground by the creek side and took some rock out which yielded nothing but blanks. He seems then to have become discouraged, and soon afterwards died, being over 80 years of age. His assistant went to Mexico, where all trace of him was lost.

I have given the story thus far somewhat in detail, as it illustrates so well the characteristic features of the deposit, and the experience of every one who has worked with it. The details were given me personally by Mr. Harralson. As this occurred about the time of the gold discoveries in Alaska the particular spot where the platinum was found was called the Klondike, which name it bears to this day.

A few years after this the matter came to the attention of Dr. C. D. W. Colby, then of Dillsboro, Jackson county, N. C., now of Asheville, who has been since that time its most earnest and persistent advocate. Much credit is due him in this matter. Starting, as he himself states, with the very elementary knowledge of platinum possessed by the average practicing physician, and with the still more elementary facilities at his disposal in a mountain village of western North Carolina, he was able to satisfy himself that the rock contained platinum, and in what he believed to be paying quantities.

Following are the results of certain assays made for Dr. Colby by a Mr. Jenkins, chemist for a copper company operating in the western part of North Carolina.

3.27	0.65	1.31	0.65
1.31	1.31	trace	0.71
3.73	3.85	0.16	trace
3.60	3.76	0.24	
3.92	0.71	0.32	

These assays were all made by the wet method, and in addition to these figures many blanks were obtained. Dr. Colby also obtained the following figures, together with a number of blanks, from well-known professional assayers:

F. F. Hunt, New York.....	2.6
Trubeck .....	3.1
Ledoux and Co. ....	3.07
G. C. Childress, Knoxville.....	1.0

Dr. Colby then began a most discouraging nine-year campaign of promotion, bringing the matter to the attention of practically every large platinum-working concern in this country, and last of all to the concern with which I am connected.

It was the same story everywhere. His tale was listened to with more or less interest, the samples were turned over to the company's chemist, who invariably reported "no platinum." Then a letter, not always too polite, closed the negotiations in that direction.

Dr. Colby, having several times been able to correct professional assayers to whom the examination of the rock had been intrusted as to the proper handling of a silver bead containing platinum, became convinced that the trouble lay with the chemists; that the platinum existed in the rock in some new form, which required a special method of analysis to detect it.

In the course of the campaign the matter was brought to the attention of the United States Geological Survey, and Dr. Day and Mr. Sterrett visited the ground. A car load of rock was taken out and tested on one of the concentrating tables which had proved very successful on the black sands of the Pacific coast. The examination of the concentrates showed no platinum. Dr. Colby had some experiments made with dry concentrators, which he says gave fairly good results. No figures on these results were furnished us.

The matter came to our attention early in the year 1911. The first thing to be done in the matter was to write to the Geological Survey.

The reply of the Survey stated that nothing had been published on the subject; that the rocks in the vicinity were hornblende schist and sericite quartz schist, which might represent metamorphosed phases of volcanic rock such as andesite and rhyolite; that it was claimed that the sulphides in the hornblende schist carried platinum in some form difficult to extract; and that the Survey had not determined this point.

With this curious history of the situation laid fully before us we were inclined to place considerable confidence in Dr. Colby's hypothesis of a new form of occurrence of platinum. Bearing in mind that the properties of compounds in the native state are often very dif-

ferent from their properties when artificially produced (*e. g.*, the insolubility in acids of certain native oxides of iron, and the comparative insolubility in aqua regia of sperrylite), we thought it not unreasonable that we might have to deal with a new compound of platinum of peculiar properties, and in the examination of the rock we were careful to follow Dr. Colby's directions literally, however useless it might seem.

The first sample submitted by Dr. Colby weighed about five pounds. One third of it was retained for examination in our laboratory, and the remainder, in lump form, was sent to Dr. Harry F. Keller, of Philadelphia, who ground it, divided it into two portions, retained one himself, and sent the other to Mr. Whitfield of the laboratory of Booth, Garrett and Blair. The portion analyzed at our laboratory was decomposed by aqua regia, according to Dr. Colby's directions, and yielded the surprising figure of eight ounces of platinum and two ounces of iridium to the ton. Dr. Keller and Mr. Whitfield decomposed the rock by hydrofluoric acid and Dr. Keller found six tenths of an ounce of platinum to the ton, while Mr. Whitfield found nothing.

Mr. Eldred, of our company, then visited the ground, took his own samples and brought them north in his hand satchel. Two assays on these samples, by the fire method according to Dr. Colby's directions, gave 0.8 and 1.1 ounces per ton platinum.

The services of an experienced mining engineer, Mr. James W. Neill, of Pasadena, Cal., were then called into requisition. Mr. Neill visited the ground with Mr. Eldred, and took samples from eight localities. The assays of these, by the fire method, gave the following figures:

0.2	0.3	0.2	trace	1.25	0.4
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and two blanks.

I will not take the time to go over in detail all the assays that were made on the property. Suffice it to say that over fifty assays were made in our laboratory, a few of which were blanks, a larger number gave perceptible traces, and a smaller number ran anywhere from half an ounce to one ounce per ton. Nothing approaching the figure given by the first sample was ever found again.

At an early stage of the assaying work it was recognized that to carry out such work in the laboratory of an industrial works devoted to the working and refining of platinum on a large scale was a matter of some delicacy. The obvious way to guard against infection was to run blanks, and this was done with perfect success for eight months. At the end of that time the furnace in which the assays were made began to show signs of infection from the fine platinum dust that might be detected almost anywhere in the works, and the blank assays began to give minute traces of platinum. A new furnace was then installed in another room, and satisfactory blanks were again obtained. It was not considered prudent to do much in the way of sending out samples to other laboratories for check analyses, but such check assays were made in our own laboratory by Dr. Harry F. Keller, of Philadelphia, and the results agreed quite well with our own figures. As an illustration of the care that was taken to avoid false results, the air supply of the furnace was examined. The furnace was fired by gas mixed with air supplied from a blower in a distant room. This blower supplied air to all parts of the establishment, and its intake was not above suspicion. A glass tube filled with cotton wool was fastened to one of the air cocks and the air allowed to blow through it for two weeks. The cotton was then burned and the ash examined for platinum. None was detected.

The examination of the ground was carried out by our mining engineer, Mr. James W. Neill, with great thoroughness. Samples were taken over an area of six or seven square miles in the immediate vicinity of the Klondike, and the peculiar rock of the region was traced fifteen miles north, to Danville, Virginia, and fifty miles south, to a point near Cedar Falls, N. C., and traces of platinum were occasionally detected in it. The work lasted a year, and was completed by an examination of the watershed of the region for placers. This latter piece of work was done by Mr. John A. Ritter, and extended far and wide, in the case of one river to a point two hundred miles from Ruffin. In this examination traces of platinum were found in the sands of the James river two miles west of Richmond; in the Dan river, where it is joined by Hogan's creek, which drains the Ruffin region; in the Dan river at Danville, Va.; in the

Roanoke river at Weldon, N. C.; and in the creek bed on the property at Ruffin. The richest sample (and the only one yielding enough platinum to weigh) was from the mouth of Hogan's creek. It contained about forty-five cents' worth of Pt per ton of gravel. Another sample taken only a few yards away was blank. This was the uniform experience wherever Pt was found, pointing to the occurrence of the metal in sparsely scattered single particles. In all other cases where Pt was found it was merely as an iodide reaction.

Traces of gold were found at the mouth of Hogan's creek and in the Dan river near Clarksville, Va.

But I take it it is of less interest to the present gathering to learn that the platinum deposit of North Carolina is commercially unimportant than to learn something about the nature of the rock that carries the Pt, and the form in which the metal occurs.

As you see by the samples the rock is evidently sedimentary in its origin. It has a density of 3.03. On roasting it becomes so friable that it may readily be reduced to its grains by the fingers. An analysis of the principal constituents of the rock, made at the Massachusetts Institute of Technology, gave the following figures:

SiO <sub>2</sub> .....	50.01
CaO .....	9.02
MgO .....	4.83
Al <sub>2</sub> O <sub>3</sub> .....	15.21
Fe .....	9.66
TiO <sub>2</sub> .....	2.45

The rock also contains the alkalis and varying amounts of sulphur. It is noteworthy that it appears to contain no chromium. The sulphur is present as sulphides of iron, in two forms, soluble and insoluble in hydrochloric acid, probably the ordinary pyrrhotite and pyrite. It was Dr. Colby's idea that the sulphides carried the platinum. Our experience negatives that. The samples richest in Pt were often poor in sulphides. Occasionally large crystals of sulphides would be found, and an assay of the hand-picked sulphides showed no platinum.

In order to determine, if possible, the form in which the platinum occurred in the rock about 150 assay tons of rock were coarsely



crushed and then ground to fine powder in rolls, with the idea of finely dividing the rock without reducing the size of the metallic particles, if any such were present. The ground mixture assayed 0.4 ounces per ton Pt, which would mean 60 milligrammes of metal in the lot. Concentration was effected by very gentle washing in the following manner: the sand was placed in a large jar, and a small stream of water introduced by a tube reaching to the bottom of the jar, and the water allowed to overflow until it ran perfectly clear. The residue, which amounted to about one third of the original amount, was dried and put through the rolls again, afterwards being washed in the same manner as before. After several repetitions of this process there remained a few grams of material, which was pulverized by hand in an agate mortar filled with water, a little at a time. The very fine material thus resulting was panned off from time to time and there remained finally a surprisingly large amount of flattened metallic particles. On digestion with hydrochloric acid the greater part of these dissolved with the familiar odor which showed them to be particles of steel derived from the grinding machinery. There remained 13.8 mg. of bright metallic particles. These were not attacked by dilute nitric acid. On heating to redness on the lid of a porcelain crucible many of the particles preserved their luster unchanged, and some of them turned a steel blue without loss of luster. A few particles showed the change of color over a part of their surface, suggesting palladium. (Samples.)

The quantity of material was rather small for an analysis, but the attempt was made. The particles were fused with zinc for an hour and the button dissolved in hydrochloric acid. The fine black residue was treated with aqua regia diluted with four volumes of water for a long time, and there remained a small black residue which was probably iridium. It weighed 0.4 mg., and was unaltered in appearance by ignition. The solution was evaporated to dryness and taken up with 60 per cent. alcohol and solid  $\text{NH}_4\text{Cl}$ . A small brick-red precipitate remained. On ignition the mixed Pt and Ir sponge weighed 7.7 mg. The alcoholic filtrate was yellowish, and gave a reaction for iron. This is significant in connection with the fact that the particles had withstood both hydrochloric and dilute

nitric acids. On warming the alcoholic filtrate it turned a deep brown color, again suggesting palladium. From the filtrate 2.7 mg.  $\text{Fe}_2\text{O}_3$  were obtained by precipitation with ammonia.

The 7.7 mg. of Pt-Ir sponge was treated with dilute aqua regia until a constant residue was obtained, which weighed 1.8 mg. Adding this to the 0.4 mg. previously obtained, we have about 16 per cent. Ir. The Pt figures about 40 per cent. and the Fe 15 per cent., leaving 29 per cent. unaccounted for. No further evidence of Pd could be obtained from the very small amount of material.

The total recovery of acid-resisting metallic particles by this method was only about 25 per cent. of the assay value of Pt, and undoubtedly comprised only the largest particles. The term largest is to be understood in a relative sense only, as none of the particles could have weighed as much as 0.1 mg.

There seems to be no doubt that the platinum exists in the rock in the form of the usual alloy or mixture of the different platinum metals and iron, with probably a greater proportion of iridium than is usual in the Russian variety. It is worthy of note that as far as is shown by the various published analyses of platinum from American sources these ores have a greater iridium content than the ores from Siberia.

As an experiment on a larger scale, half a ton of rock from a locality about two miles distant from the Klondike was smelted in the experimental blast furnace at the Massachusetts Institute of Technology, and yielded 11 mg. of platinum. A sample of this rock had given an assay value of 0.4 ounces per ton, accompanied by a satisfactory blank. A second assay had yielded nothing. It was customary to make assays on 4 A. T. lots of ore.

All the foregoing facts are consistent with the hypothesis that the platinum exists in the rock in sparsely scattered granules, so few in number that by no amount of rolling and mixing can we bring the sample into such a state that there will be at least one such granule in each assay ton of the ore.

As a matter of interest, during the progress of the assaying work on the North Carolina rock, we were led to examine samples of rock from a great many localities for platinum. The curious fact

was discovered that it was almost impossible to obtain a blank when the rock was largely ferruginous, while on rocks composed mainly of quartz there was no difficulty in this respect. In no case was anything more than the extremely delicate iodide reaction obtained. In applying this test it is important to note that it is interfered with by the presence of iron, nitric acid, and alcohol. The latter substance is likely to be the most frequent cause of failure to obtain the reaction. In certain ferruginous rocks, when the point was reached where the platinum, if present, should remain insoluble on the watch glass after taking up the sal-ammoniac with 60 per cent. alcohol, there was often an almost microscopic trace of a white residue. Performing the filtration by faith, and washing with 95 per cent. alcohol, also by faith, the critical point of the procedure was reached. If the supposed precipitate was now dissolved off the filter by hot water, no iodide reaction could be obtained; but if the filter was first dried until all odor of alcohol had disappeared, an iodide reaction could often be obtained. In this way reactions were obtained from certain building stones from Lower Merion township, Montgomery county, Pa., and from a trap dyke in the neighborhood of our laboratory in Westchester county, N. Y. The sands of the Bronx river, a small stream in our vicinity, gave no reaction when unconcentrated, but when concentrated by hand panning a reaction could be obtained. Bearing in mind the relation of platinum to the iron group, and the fact that iron always accompanies platinum, it is not so surprising that platinum should occur in small traces with iron wherever the latter is found.

We also examined the rocks near Sassamansville, Berks county, Pa., which are mentioned in Kemp's report as giving irregular indications of Pt, and found iodide traces in several samples. To judge by the depth of color, these samples were not as rich as certain of the Lower Merion building stones.

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